Efficacy of Various Organic Solvents for the Rapid Direct Determination of Moisture in Meat and Meat Products

By EDWARD H. COHEN[1] and CHARLES P. KIMMELMAN (Eastern Marketing and Nutrition Research Division, Agricultural Research Service, U.S. Department of Agriculture, Philadelphia, Pa. 19118)

Twenty-seven organic solvents that form azeotropes with water were tested for use in shortening the time required for the determination of moisture in ground meat, processed frankfurters, and uncooked pork sausage mixture. A goal of 15–30 min was sought with a minimum of 95% recovery of the available moisture content in the meats by use of inexpensive "in stock" available equipment that could be used by nontechnical personnel. Azeotropic distillation in a modified 10 ml Bidwell-Sterling moisture receiver, West condenser, ⅌ 40/50 boiling flask, and matching enclosed heating mantle was used for periods of time up to 30 min. For example, in 2 lots of ground meat after a 30 min distillation time, the following average recoveries (%) were found: toluene 95, octane 97, nonane 100, xylene 99.7, ethylbenzene 99.4, cumene 100.3, 2-ethyl-1-hexanol 97.7, 1-heptanol 99.5, 1-octanol 99.8, 2-octanol 100.1, and butyl ether 98.5. The latter 9 solvents were found to give acceptable recoveries in 15 min. Chlorinated hydrocarbons were also evaluated but are not recommended due to toxicity levels and the need for specialized equipment.

A previous study (1) of rapid moisture analysis in meat described the broad spectrum of methods available to the meat industry and food scientists. Methods proposed (IR oven, IR balance, and "hot plate" method) were inexpensive, fast, reproducible, and accurate within the conditions sought for a rapid method. During the study the author found that azeotropic distillation using toluene fit most of the above criteria with the exception of reduced time of analysis and a lower recovery. The results of a 30–60 min distillation gave moisture recoveries 3–5% lower than the official first action method, 24.003(a) (2). It was decided to investigate readily available water-insoluble azeotrope-forming organic solvents which could be used to determine moisture directly.

As no exact tolerances have been established by the AOAC for moisture analysis, this laboratory has endeavored to approach guidelines set up by the USDA Consumer and Marketing Service of a tolerance within 1% for moisture (3).

Experimental

Apparatus and Reagents

(a) *Distillation apparatus.*—Flat-bottom 250 ml flask with ⅌ 40/50 joint, 40/50 to 24/40 ⅌ reducing adapter, modified Bidwell-Sterling 10 ml distillation receiver, 30 cm full-jacketed West condenser, and Electrothermal MG 7803 heating mantle regulated by variable transformer. Adapter reduces interference from foaming and raises moisture receiver above heating mantle. (Heating mantle has full upper insulation for above flask and will fit any 250 ml size flask. Use asbestos cloth for further thermal insulation of exposed glassware up to condenser.)

(b) *Reagents.*—*See* Table 1.

(c) *Samples used.*—Commercial ground beef, frankfurters, and uncooked sausage mixture.

Preparation of Sample

Grind sample 2 times (except once for ground beef) through ⅛″ plate, then once through 5/64″ plate at 3°C, using prechilled grinder.

Determination

Place 10.00±0.05 g meat product in boiling flask and quickly add 100.0±0.5 ml solvent to prevent evaporation of water from sample. Assemble apparatus. Adjust heat to produce reflux level of 2″ above tip of condenser. Time to reach boiling is ca 4–6 min, depending on bp of solvent (*see* Table 1). Lower voltage if foam, due to presence of fat, rises above adapter. Read volume of water distilled at bottom of meniscus of solvent-water interface. Estimate

[1] Present address: Surveillance Division, Environmental Protection Agency, Philadelphia, Pa. 19106.

Please address requests for reprints to Meat Laboratory, Eastern Marketing and Nutrition Research Division, Agricultural Research Service, U.S. Department of Agriculture, Philadelphia, Pa. 19118.

volume to ±0.02 ml. Take volume reading at time solvent first appears in receiver and then record volume at 15 and 30 min. For 10 g sample, % moisture = ml water × 10. Conduct all distillations in well ventilated hood (>150′/min velocity at 1′ opening), using great care with highly flammable and/or toxic solvents (*see* Table 1).

Results and Discussion

A study of the physical characteristics of azeotrope-forming organic solvents was conducted to determine those solvents best suited for use in the rapid direct determination of water in meat products. Listed in Table 1 are 27 solvents; however, only 18 solvents were actually tested with meat products. Benzene and ethyl ether were not used because of high solubility of water in the solvent and low per cent water in the azeotrope. The latter property tends to lengthen the time required for water removal. Seven of the 9 chlorinated hydrocarbons considered were not tested due to the low per cent of water in the azeotrope, high toxicity values, high solubility of water in solvent or solvent in water, or lack of azeotropic data.

Table 2 shows the amount (ml) of moisture distilled azeotropically during a 15 min and a 30 min period for 2 separate lots of ground beef, one lot of processed frankfurters, and one lot of uncooked sausage mix. The results are based on 2 separate distillations of 10 g samples from the same lot. The recoveries, based on the values obtained by the official AOAC method, are calculated only for the 30 min distillation interval.

With a goal of achieving 95% recovery of moisture within a 30 min distillation period, a number of solvents show promise for use in the rapid analysis of meat products. The recoveries (per cent) achieved with these solvents were as follows: octane 96.8–98.4, nonane 99.4–100.5, ethylbenzene 99.1–100.0, cumene 100.1–100.6,

Table 1. Physical characteristics[a] and other properties of organic solvents tested for the rapid direct determination of moisture in meat and meat products

Solvent	bp at 760 mm, °C		H_2O Azeotrope, %	Solubility, %		sp gr	Fire Hazard	Toxicity TLV, ppm	Approx. Cost,[b] $/Gal.
	Solv.	Azeotrope		Solv. in H_2O	H_2O in Solv.				
Pentane	36.1	34.6	1.4	0.04	0.05	0.626	Yes	1000	3
Hexane	69.0	61.6	5.6	0.04	—	0.660	Yes	500	2
Heptane	98.4	79.2	12.9	0.01	0.02	0.683	Yes	500	2
Octane	125.7	89.6	25.5	0.02	0.01	0.704	Yes	500	40
Nonane	150.8	95.0	39.8	insol.	insol.	0.717	Yes	—	36
Cyclohexane	81	69.8	8.5	0.01	0.01	0.779	Yes	300	10
Ethylbenzene	136.5	92.0	33	0.02	0.05	0.867	Moderate	100	6
Cumene	152.4	95.0	43.8	0.01	0.05	0.862	Moderate	50	6
2-Ethyl-1-hexanol	185	99.1	80.0	0.10	2.60	0.834	Moderate	—	4
1-Heptanol	176	98.7	83.0	0.09	0.02	0.822	Moderate	—	33
1-Octanol	195.2	99.4	90.0	—	0.03	0.825	—	—	11
2-Octanol	178.5	98.0	73.0	<0.05	<0.10	0.819	Moderate	—	11
Benzene	80.1	69.4	8.9	0.08	0.06	0.879	Yes	200	6
Toluene	110.6	85.0	20.2	0.06	0.05	0.866	Yes	200	4
m-Xylene	139.1	94.5	40.0	0.05	0.05	0.864	Yes	100	7
Ethyl ether	34.6	34.2	1.2	7.5	1.30	0.714	Yes	400	5
Butyl ether	142	94.1	33.4	0.02	0.03	0.784	—	—	10
Benzyl methyl ether	174	97[c]	—	insol.	—	0.987	—	—	450
Tetrachloroethylene	121	88.5	17.2	<0.01	0.02	1.627	No	100	8
Trichloroethylene	86.7	73.0	—	0.10	0.02	1.466	No	100	5
cis-1,2-Dichloroethylene	60.1	55.3	3.4	0.04	0.77	1.291	No	200	90
trans-1,2-Dichloroethylene	48.4	45.3	1.9	0.03	0.63	1.265	No	—	70
Tetrachloroethane	u^d-130.5 s^d-146.3	—	—	0.32	0.03	1.588 1.600	No	5	8
1,1,1-Trichloroethane	74.1	—	—	0.13	—	1.325	No	350	6
1,1,2-Trichloroethane	113.7	86.0	16.4	0.05	0.45	1.443	No	10	9
1,1-Dichloroethane	57.3	—	—	0.55	—	1.174	No	100	12
1,2-Dichloroethane	84	71.6	8.2	0.80	0.16	1.257	No	50	5

[a] See refs. 4–7; TLV = total lethal volume.
[b] Lowest grade readily available.
[c] Azeotropic bp determined by this laboratory.
[d] *u* = unsymmetrical, *s* = symmetrical.

Table 2. Recoveries of moisture in meat products by direct azeotropic distillation in various organic solvents, using short time intervals[a]

	Ground Beef,[b] Moisture Recd After				Frankfurter, Moisture Recd After				Sausage, Moisture Recd After			
	15 min	30 min			15 min	30 min			15 min	30 min		
Solvent	ml	ml	sd[c]	%[d]	ml	ml	sd	%	ml	ml	sd	%
Pentane	trace	0.11	0.05	1.9								
Hexane	0.80	1.48	0.11	25.3								
Heptane	3.61	4.88	0.25	83.6								
Octane	5.28	5.67	0.12	97.2	5.10	5.37	0.12	98.4	4.10	4.45	0.14	97.9
	4.82	5.15	0.21	96.8								
Nonane	5.66	5.80	0.02	99.4	5.39	5.45	0.00	99.8	4.42	4.53	0.02	99.7
	5.25	5.35	0.00	100.5								
Cyclohexane	1.35	2.23	0.18	38.2								
Ethylbenzene	5.75	5.81	0.01	99.6	5.25	5.46	0.06	100.0	4.38	4.53	0.04	99.7
	5.02	5.28	0.11	99.1								
Cumene	5.78	5.84	0.05	100.1	5.40	5.53	0.04	101.3	4.45	4.57	0.01	100.6
	5.35	5.35	0.07	100.5								
2-Ethyl-1-hexanol	5.72	5.72	0.01	98.1	5.35	5.35	0.01	98.0	4.33	4.41	0.08	96.9
	5.18	5.18	0.04	97.3								
1-Heptanol	5.79	5.80	0.01	99.4	5.40	5.40	0.00	98.9	4.38	4.40	0.03	96.8
	5.20	5.30	0.00	99.6								
1-Octanol	5.94	5.98	0.32	102.5	5.43	5.46	0.04	100.4	4.50	4.50	0.00	99.0
	5.25	5.29	0.05	99.3								
2-Octanol	5.83	5.87	0.09	100.5	5.45	5.43	0.11	100.4	4.55	4.55	0.00	100.1
	5.30	5.30	0.00	99.6								
Toluene	4.85	5.49	0.19	94.1	4.80	5.23	0.04	95.8	3.95	4.30	0.21	94.6
	4.48	5.07	0.26	95.2								
m-Xylene	5.23	5.35	0.07	100.5	4.76	5.34	0.09	97.8	4.48	4.54	0.02	99.9
	5.68	5.77	0.09	98.9								
Butyl ether	5.80	5.84	0.02	100.1	5.20	5.33	0.18	97.6	4.25	4.50	0.00	99.0
	4.92	5.15	0.28	96.8								
Benzyl methyl ether	5.05	5.43	0.11	102.0								
Tetrachloroethylene	5.75	5.75	0.00	98.6								
1,1,1-Trichloroethane	—	3.74	—	63.7								

[a] Results listed as ml water. To obtain % moisture multiply × 10.
[b] Two lots of ground beef used.
[c] sd = Standard deviation, n = 2.
[d] % Recovery = [% moisture (rapid method)/% moisture (official method)] × 100.

2-ethyl-1-hexanol 97.1–98.1, 1-heptanol 96.8–99.6, 1-octanol 99.3–100.4, 2-octanol 99.6–100.4, toluene 94.1–96.4, *m*-xylene 97.8–100.5, butyl ether 96.8–100.1, benzyl methyl ether 102.0, and tetrachloroethylene 98.6. Solvents that did not recover 95% of the moisture found in ground beef were not used for the second lot of ground beef or for frankfurters and sausage. The chlorinated hydrocarbons were not used further because of technical problems. The special moisture receivers available for use with solvents with a specific gravity greater than 1.000 were not calibrated to the specific gravity of the specific solvents used; hence, accurate readings were not possible. When ordering such custom-made receivers the specific gravity of the solvent to be used must be designated.

Of the 13 solvents mentioned in the analysis of ground beef several are not recommended for general use. These are: tetrachloroethylene, which requires custom-made receivers; octane, which is slightly more expensive than nonane and gives lower results; 2-ethyl-1-hexanol, which shows good recovery but has a disadvantage in that it dissolves 2.6% water; 1-heptanol, which is less accurate and more expensive than 1- and 2-octa-

nol; toluene, which gives lower recoveries than *m*-xylene; and benzyl methyl ether, which has no advantage over less expensive solvents. Among the remaining solvents nonane is the most expensive; the rest are within the same price range. Butyl ether is considered dangerous, as it will form explosive peroxides, especially when anhydrous. Although nonane, cumene, and 1- and 2-octanol gave lower recoveries at 15 min than at 30 min analysis, they may be used for all 3 meat products with a minimum of 97% recovery.

The results reported are uncorrected for the following sources of error, many of which compensate one another:

(*a*) Glassware calibration using a 5.00 ml class A pipet at 20°C. The 5 ml graduation on the moisture receiver was 4.966±0.030 and the 9 ml graduation was 8.957±0.046, averaged for 9 moisture receivers.

(*b*) Error in water expansion due to temperature. Volumes were read at 26°C and uncorrected for expansion. The volume of expansion of 5.000 ml water from 20 to 26°C is +0.0045 ml.

(*c*) Readability of the meniscus shape due to the effect of different solvents.

(*d*) Absolute recovery of water in the particular apparatus used.

(*e*) Solubility of water in the particular solvent and solubility of the solvent in water.

(*f*) Cleanliness of glassware.

(*g*) Loss of water through the condenser.

The main consideration of the above sources of error is that if a rapid method is calibrated to an official method and produces consistent (repeatable) results, then, using the same conditions of temperature, apparatus, solvents, etc., the errors may be disregarded.

The reduction in time from several hours for the official method of moisture analysis to 15–30 min demonstrated above warrants further investigation by a collaborative study of the distillation methods as an alternative rapid analysis for moisture in meat products.

References

(1) Cohen, E. H. (1971) *JAOAC* 54, 1432–1435

(2) *Official Methods of Analysis* (1970) 11th Ed., AOAC, Washington, D.C.

(3) Fried, I. (1969) *Nat. Provisioner* **161**, 32–33, Nov. 15

(4) Dow Chemical Co. (1952) *Azeotropic Data, Advances in Chemistry Series*, No. 6, American Chemical Society, Washington, D.C.

(5) Marsden, C. (1963) *Solvents Guide*, Cleaver-Hume Press Ltd., London

(6) Hodgman, C. D. (ed.) (1962) *Handbook of Chemistry and Physics*, 44th Ed., The Chemical Rubber Publishing Co., Cleveland

(7) Sax, N. I. (1968) *Dangerous Properties of Industrial Materials*, 3rd Ed., Reinhold, New York